

EUROPEAN FEDERATION OF CHEMICAL ENGINEERING  
WORKING PARTY ON DISTILLATION, ABSORPTION AND EXTRACTION

STANDARD TEST SYSTEMS FOR LIQUID EXTRACTION

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2nd edition

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## FOREWORD

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The Working Party on "Distillation, Absorption and Extraction" is one of the working parties set up by the European Federation of Chemical Engineering, a non-profit making association of technical and scientific societies which are active in the fields of Chemical Engineering, Chemical Technology and Process Engineering. The main activity of the Working Party on "Distillation, Absorption and Extraction" is directed towards organizing informal discussion meetings on subjects within its terms of reference; occasionally, the Working Party also participates in large international conferences, e.g. the International Symposia on Distillation in 1969 and 1979.

Progress in the field of separation techniques and associated equipment development very much depends on experimental investigations. During the Working Party's discussion meetings, it was soon concluded that the situation with respect to the use of test mixtures for determination of the performance of equipment was less than satisfactory. As a consequence, a decision was made to attempt to standardize test mixtures for distillation columns. As a result of a joint effort, the manual "Recommended Test Mixtures for Distillation Columns" was issued in 1969. This latter work mainly collates data from the literature and presents the information required for description of some ten test mixtures.

Following publication of the distillation manual, standardization of test mixtures for liquid-liquid extraction studies was initiated. A Sub-Committee of the Working Party was set up in 1972 with Dr. T. Misek (Czechoslovakia) as the Chairman, and Dr. H.W. Brandt (West Germany) and Professor C. Hanson (Great Britain) as members.

The situation regarding the proper choice of systems for liquid-liquid extraction studies was found to be even more confusing than that for distillation. Literature data on equilibria and physical properties were conflicting; thus indicating the need for experimental verification and completion of relevant physical data. After due consideration, the Sub-Committee selected three test mixtures for closer investigation. It is most gratifying that the Sub-Committee was able to secure the cooperation of universities and industrial laboratories for determination of the necessary data. A list of contributors is presented. The preface describes the general principles upon which the work performed was based.

The preparatory work of data collection took considerably more time than originally planned and the collation of these data also proved to be time-consuming. Now that the work is complete, the Working Party is very pleased to present with

this manual "Recommended Systems for Liquid Extraction Studies", the results of the study of which so many people contributed in various ways.

Special thanks have to be extended to Dr. T. Míšek and the other members of the Sub-Committee for their enthusiasm and perseverance in completing this project.

I would also like to gratefully acknowledge the contribution of Ing. L. Strnadová, Ing. D. Stangl, and their co-workers who assisted Dr. Míšek in assembling and treating the experimental data and in the preparation of the manual.

October 1977

The first edition of the manual "Recommended Systems for Liquid Extraction Studies" has been sold out very quickly. This is gratifying, in particular because it indicates that the work of the Sub-Committee in preparing the manual has been very rewarding. It is therefore with pleasure that I can now announce a second edition of the booklet, in which much of the positive criticism, received on the first edition, could be taken into account. The Working Party has again been lucky to obtain the cooperation of a small Sub-Committee of experts, this time with as members Dr. T. Míšek (chairman), Dipl.-Ing. R. Berger and Ing. J. Schröter. Their efforts are gratefully acknowledged, as well as the support given to the project by the BASF and the Bayer Companies.

September 1984

Professor F.J. Zuiderweg,

Chairman of the Working Party  
on Distillation, Absorption and  
Extraction

## PREFACE TO THE FIRST EDITION

The period of the last three decades has been characterized not only by rapid development in the use of liquid extraction in industry but also by associated progress in research and development of extraction equipment, processes and the behaviour of two-phase liquid systems in general.

For all these purposes, it was necessary to perform experimental work using different liquid systems the number of which is still growing. Many systems have been employed, chosen according to the specific objectives of particular studies, availability at the time or other random effects. In addition to their composition, the systems used have been defined by some of their thermodynamic and physical properties. These have served as a starting point for the quantitative description of the effects of these properties on the phenomena studied in two-phase liquid systems.

However, it has become apparent that this approach has serious drawbacks. Thus physical and thermodynamic properties chosen are not able to define completely the systems used; systematic errors and deviations are often introduced in their measurement, while formerly unknown property effects and phenomena are revealed at later stages making the research work performed with different systems uncomparable or even useless. When testing extraction equipment, the use of different systems leads necessarily to different results and to conclusions that may not be generally justified.

The recommendation of using only a few selected systems in liquid extraction studies is aimed at providing a comparable basis for these studies, and at achieving deeper knowledge of these systems, so that the above disadvantages can be avoided. Thus, in addition to testing extraction equipment in the laboratory and pilot plant, the recommended systems should also be used for studying the basic components of the extraction processes, e.g. formation and motion of drops, transfer of heat and mass, coalescence, interphase phenomena, etc. Hopefully, the work of different authors could then become comparable and the explanation of anomalous phenomena, deviations and strange behaviour will be possible.

The Sub-Committee of the Working Party faced a difficult task in selecting the systems which should be considered and eventually recommended for general use after thorough investigation. The selection necessarily involved compromises and the systems chosen for this study are not ideal. However, they can undoubtedly meet the objective and are supported by the experience of the principal institutions dealing with liquid extraction in Europe, the data in the literature and the results of the contributors obtained during this project.

The following systems were chosen for thorough investigation:

1. toluene - water - acetone
2. n-butanol - water - succinic acid
3. methyl isobutyl ketone - water - acetic acid.

For practical reasons they are all aqueous but they do differ in the most significant physical property for two-phase liquid systems - interfacial tension. Systems (1) and (2) are characterized by distribution coefficients approaching unity and constant over a wide concentration range. They may be assumed to involve physical extraction in contrast to System (3) which, while being widely used in practice, show some anomalous behaviour. System (1) represents an essentially immiscible basic binary system, whereas in Systems (2) and (3) the basic binaries are mutually soluble. The characters of Systems (1) and (3) provide very different wettability of different solids by both phases, whereas in System (2) these differences are smaller. Analysis of all the systems chosen is simple; the results are reproducible and all the systems consist of easily available raw materials.

These arbitrarily chosen characteristics of the selected systems do not mean that it would be impossible to find other suitable or even more suitable systems. However, selection, description and introduction into practice would present difficulties which cannot easily be overcome at the present time.

This publication presents results of the work of a wide group of authors and is aimed at engineers as well as scientists dealing with extraction systems. Hence, an arrangement was accepted which makes possible utilisation of the data measured either for technical purposes in the form of a smoothed analytical function or in the original form for deeper studies. For the most part, the data have been measured by two or more contributors, using different methods and raw materials from different sources. The authors believe that this published set of data will encourage the search for a deeper interpretation on a physico-chemical basis.

The information on fire and safety properties and the behaviour of selected systems has been included for guidance only; the reader should check the appropriate national regulations, the observance of which is obligatory.

The text of the work has been kept as brief as possible. More detailed data on the measurements can be obtained from individual authors, whose names and addresses are presented.

October 1977

Ing. T. Míšek, DrSc.

Chairman of the Working Party  
Sub-Committee

## PREFACE TO THE SECOND EDITION

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The first edition of this booklet was sold out astonishingly quickly, although its subject is of direct interest only to a relatively small circle of specialists. In preparing the new edition the editors were concerned to make use of lessons learned from the first edition, to adopt uniform nomenclature, and to restrict the Appendix to those tables that are directly relevant to extraction processes. As far as the arrangement and presentation of the experimental results are concerned, the concept of the first edition has been retained.

Considerable alteration was found to be necessary concerning the choice of the systems. The original aim of the project was to choose systems with widely different interfacial tensions. To represent the medium range of interfacial tension, the system methyl isobutyl ketone - water-acetic acid was chosen, but in the course of investigations it was found that in this system mass transfer is accompanied by interfacial turbulence and that chemical interactions take place. Both effects lead to a considerable increase in the rate of mass transfer. Because of this abnormal behaviour, the system cannot be taken as representative for physical extraction and therefore can not be recommended for investigations involving mass transfer. Already the first edition includes in the "Concluding Recommendations" (page 17) a corresponding reference. For this reason the editors have decided, after careful consideration, to exclude the data of the system methyl isobutyl ketone-water-acetic acid from the second edition.

Not only from an industrial point of view there was an urgent need for a system with medium interfacial tension that could be used for investigations involving mass transfer, since most solvents used industrially have interfacial tension in this region. In searching for a suitable system various ethers, esters, and ketones were considered. The final choice has been the system butyl acetate-water-acetone. The measurements were carried out in accordance with the general principles for the test-system project described in the preface to the first edition.

Five years after publication of the first edition it is possible to report on some results achieved by the test-system project. To a considerable extent the hopes expressed in the preface to the first edition have already been fulfilled. For instance, several cases in which equipment investigation revealed phenomena that were very difficult to explain have been well interpreted by means of detailed scientific investigations on droplets, so that in this way fundamental research at universities and industrial research were usefully complemented. At test runs at different companies the test systems have provided a non-competitive basis for exchange of information, division of tasks, comparisons, and better choice of equipment for specific applications. In this way test systems were promoting co-operation between a number of



research centres. Finally, the existence of test systems with reliable data relating to equilibrium and other physical properties, coupled with detailed methods of measurement, promoted new research projects in the field of extraction and research into liquid-liquid systems themselves.

In addition it is hoped that the existence and further application of the Recommended Systems will promote better mutual understanding between research workers and technicians in the field of liquid extraction.

In a time of economic recession the economic aspects of such cooperation ought not be underestimated. The joint effort of many research centres, coordinated by the EFCE Working Party, presents thus a useful and practical tool for both applied and academic research, which can substantially reduce the demand for work and money. With this in mind the second edition is being produced.

The editors would like to express their thanks to M. Slater and G. Orthöfer, who have undertaken the difficult task of correcting the manuscript.

July 1984

R. Berger  
T. Mišek  
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This publication originated from the joint work of many European experts. It would be invidious to differentiate between their contributions and so they are listed alphabetically:

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